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(54) Title: ENHANCED ZINC CONTAINING ORAL COMPOSITION

(57) Abstract

An improved oral composition, such as a toothpaste, gel dentifrice, toothpowder, tooth hardener, anticalculus composition, gum or lozenge, comprising a bacteriostatic and anticalculus effective amount of zinc ions and an antimicrobial effective amount of one or more essential oils is disclosed. The oral composition has longer-lasting breath freshening activity. The preferred source of zinc ion is zinc chloride. The preferred essential oils are thymol, menthol, eucalyptol and methyl salicylate.

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ENHANCED ZINC CONTAINING ORAL COMPOSITION

Field of the Invention

This invention relates to an improved anticalculus and antimicrobial effective oral composition comprising a source of zinc ion in combination with essential oils such as thymol, eucalyptol, menthol, and methyl salicylate. Oral malodor reduction is also improved with the oral composition of this invention. The oral composition of this invention is in the form of a dentifrice, mouthwash and the like.

Background

The effectiveness of zinc ions in promoting and improving oral hygiene is known. Zinc ions in oral compositions are typically provided as water-soluble zinc salts or suspensions of zinc oxide particles. Oral compositions with zinc ions have enhanced bacteriostatic activity. They also have enhanced anticalculus activity.

Essential oils are known to be germicidal. When used in oral compositions they reduce plaque, gingivitis, and oral malodor. Essential oils are aromatic compounds that are either derived from plant sources or are synthesized. Thymol is an essential oil that is well-known and widely used as an antimicrobial in oral compositions. Other essential oils include menthol, methyl salicylate, eucalyptol, anethol and eugenol. For example

LISTERINE® antiseptic mouthwash has been marketed for over 100 years, and contains thymol, menthol, eucalyptol, and methyl salicylate. More recently, essential oils have been included in formulations of dentifrice.

U.S. Patent No. 4,022,880 to Vinson et al. teaches a composition for inhibiting dental plaque and calculus comprising zinc ions and a non-toxic, organoleptically acceptable antibacterial agent.

U.S. Patent No. 4,550,018 to Ambike et al. teaches an oral composition with zinc chloride and essential oil. However, the essential oil is in a flavor amount, not an antibacterial amount.

U.S. Patent No. 4,568,540 to Asano et al. teaches an oral hygiene composition containing an effective concentration of a pharmaceutically acceptable fluoride salt, a pharmaceutically acceptable zinc salt, a specific buffering agent, a suitable vehicle. and having a pH of from about 3.5 to 6.0.

U.S. Patent Nos. 4,749,561 and 4,749,562 to Lane et al. teaches a dentifrice including a substantially water-insoluble non-cationic antimicrobial agent or a zinc salt or a mixture thereof when the dentifrice comprises at least 0.2% by weight of a lamellar liquid crystal surfactant phase.

U.S. Patent Application Serial No. 08/774,990, assigned to Warner-Lambert, teaches a stable acidic antiseptic dentifrice composition with therapeutically effective amounts of essential oils formulated at a pH of between 3.0 and 5.5. U.S. Patent No. 5,094,843 to Mazzanobile et al. teaches an anti-plaque, anti-gingivitis toothpaste with a flourine source, and a specific range of thymol, menthol, methyl salicylate and eucalyptol.

The prior art, however, does not teach an antimicrobial oral composition comprising a combination of zinc ions and essential oils wherein the bacteriostatic action of the composition is enhanced, and the oral malodor reduction activity is improved.

Summary of the Invention

It is an object of this invention to provide an improved oral composition with a bacteriostatic and anticalculus effective amount of zinc ion and an antimicrobial effective amount of one or more essential oils.

It is another object of this invention to provide an improved oral composition with longer lasting breath freshening activity.

It is another object of this invention to provide a method of making an improved oral composition containing a source of zinc ions and one or more essential oils.

It is another object of this invention to provide a method of using an improved oral composition containing a source of zinc ions and one or more essential oils.

Brief Description of the Drawing

Figure 1 is a graph of the bacteriostatic efficacy of zinc ion dentrifrice compositions made with and without essential oils.

Detailed Description

The oral composition of this invention comprises an anticalculus and bacteriostatic effective amount of zinc ion, an antimicrobial effective amount of one or more essential oils, and an orally acceptable vehicle or base .

The oral composition of this invention is preferably a dentifrice, such as toothpaste and gel. Other compositions may include mouthwashes, mouthrinses, tooth powders, tooth hardeners, antitartar compositions, anticalculus compositions, gums, tablets, lozenges, troches and the like. The base or the medium for the active components of the composition of this invention will be any which is conventionally employed and known to those skilled in the art.

The zinc ion in the composition of this invention may be provided by a number of zinc-containing compounds. The preferred source of zinc ion is one or more water-soluble zinc salts or zinc oxide. Suitable zinc salts include but are not limited to zinc sulphate, zinc chloride, zinc bromide, zinc iodide, zinc nitrate, and carboxylic acid salts of zinc. The preferred zinc salt is zinc chloride. The dentifrice composition of this invention

is formulated to provide at least about 50 ppm of zinc ion; more preferably at least about 1500 ppm of zinc ion. The water-soluble zinc salt or salts are present in the composition of this invention in an amount of up to about 5% by weight of the composition (hereinafter "w/w"), preferably about 0.25 to about 3% w/w; and most preferably about 0.5 to about 2% w/w. In the case of a dentifrice composition 0.01% zinc chloride by weight provides about 50 ppm zinc ion; 0.2% zinc chloride by weight provides about 1000 ppm zinc ion; and 0.3% zinc chloride provides about 1500 ppm zinc ion. The preferred amount of zinc chloride in the dentifrice composition of this invention is, therefore, about 0.01% w/w; the more preferred amount of zinc chloride is about 0.2% w/w; and the most preferred amount is about 0.5% w/w.

Zinc ions can also be provided by suspensions of zinc oxide particles. The zinc oxide is present in the composition in an amount sufficient to provide at least about 50 ppm zinc ion; preferably at least about 1500 ppm zinc ion.

Essential oils are volatile aromatic oils that may be synthetic or may be derived from plants by distillation, expression or extraction, and that usually carry the odor or flavor of the plant from which they are obtained. Essential oils are widely used in oral care products. Some essential oils show long-lasting germicidal effectiveness against the most common pathogens in the mouth. These pathogens are frequently associated with oral malodor, plaque, and gingivitis. The essential oils in the composition of this invention include but are not limited to thymol, methyl salicylate, menthol, eucalyptol, camphor, anethole, carvone, eugenol, isoeugenol, liminene, osimen, n-decyl alcohol, citronel, alpha-salpineol, methyl acetate, chlorothymol, citronellyl acetate, methyl eugenol, cineol, ethyl linalaol, safrola vinillin, spearmint oil, peppermint oil, lemon oil, orange oil, sage oil, rosemary oil, cinnamon oil, pimento oil, laurel oil, cedarleaf oil, and clove oil. The preferred essential oils in the composition of this invention are thymol, methyl salicylate, menthol, and eucalyptol. The most preferred essential oil is thymol.

In a dentifrice composition of this invention it is desirable that there be at least about 0.075% by weight of each of such four essential oils; preferably at least about 0.125% by weight; and most preferably at least about 0.2% by weight of each essential oil.

Thymol, also known by the chemical formula 5-methyl 2-(1-methylethyl) phenol, may be in the dentifrice composition of this invention in an amount of from about 0.01% w/w to about 1.0% w/w; preferably in an amount of from about 0.1% w/w to about 0.7% w/w; and most preferably in an amount of from about 0.2% w/w to about 0.6% w/w..

Menthol may be in the dentifrice composition of the present invention in an amount of from about 0.01% w/w to about 1.0% w/w; preferably in an amount of from about 0.10% w/w to about 0.7% w/w; and most preferably in an amount of from about 0.2% w/w to about 0.6% w/w.

Eucalyptol may be in the dentifrice composition of the present invention in an amount of from about 0.01% w/w to about 1.0% w/w; preferably in an amount of from 0.05% w/w to about 0.5% w/w; and most preferably in an amount of from about 0.07% w/w to about 1.0% w/w.

Methyl salicylate may be in the dentifrice composition of the present invention in an amount of from about 0.01% w/w to about 1.0% w/w; preferably in an amount of from about 0.04% w/w to about 0.8% w/w; and most preferably in an amount of from about 0.2% w/w to about 0.7% w/w.

The dentifrice composition of the invention may contain the following essential oils in percentages by weight: (a) thymol from about 0.01% w/w to about 1.0% w/w; (b) menthol from about 0.01% w/w to about 1.0% w/w; (c) eucalyptol from about 0.01% w/w to about 1.0% w/w; and (d) methyl salicylate from about 0.01% w/w to about 1.0% w/w.

In the preferred embodiment of the dentifrice composition of the present invention, the dentifrice composition may contain the following essential oils in percentages by weight: (a) thymol from about 0.1% w/w to about 0.7% w/w; (b) menthol from about 0.1% w/w to about 0.7% w/w; (c) eucalyptol from about 0.05% w/w to about 0.5% w/w; and (d) methyl salicylate from about 0.04% w/w to about 0.8% w/w.

In the most preferred embodiment of the dentifrice composition of the present invention, the dentifrice composition may contain the following essential oils in percentages by weight: (a) thymol from about 0.2% w/w to about 0.6% w/w; (b)

menthol from about 0.2% w/w to about 0.6% w/w; (c) eucalyptol from about 0.07% w/w to about 1.0% w/w; and (d) methyl salicylate from about 0.2% w/w to about 0.7% w/w.

The total concentration by weight of the one or more essential oils is from about 0.1% w/w to about 4.0% w/w of the dentifrice composition. In the preferred embodiment the total concentration by weight of the one or more essential oils is from about 0.5% w/w to about 3.0% w/w. In the most preferred embodiment the total concentration by weight of the one or more essential oils from about 0.8% w/w to about 2.5% w/w.

The dentifrice composition of this invention also comprises a liquid vehicle containing about 3-60% by weight of water, typically mixed with at least one humectant and a number of other ingredients. The liquid phase comprises about 20-90% by weight of the dentifrice and is generally about 25-80% liquid.

Suitable humectants in the dentifrice include sorbitol, glycerin, propylene glycol, polyethylene glycol, sorbitan, fructose, mixtures thereof and the like. Humectants, when employed, may be present in amounts of from about 10% to about 50%, by weight of the dentifrice composition.

The dentifrice also contains a gelling or binding agent as a solid vehicle agent. Gelling or binding agents which may be present include alkali metal carboxymethyl cellulose, hydroxyethyl cellulose, hydroxypropyl cellulose, xanthan, thickening silicas, Irish moss, iota-carrageenan, gum tragacanth, polyethylene glycol, polyvinyl pyrrolidone, starch and mixtures thereof.

Any suitable surface active or detergent material may also be included in the dentifrice compositions. These surfactants may be non-ionic, amphoteric, cationic, or anionic. The most preferred surfactants are anionic. These anionic surfactants include but are not limited to sodium lauryl sulfate, sodium lauroyl sarcosinate, and sodium methyl cocoyl taurate, and disodium lauryl sulfosuccinate. In the most preferred embodiment the surfactant is the anionic surfactant sodium lauryl sulfate.

Suitable nonionic surfactants in the present invention include poly(oxethylene)-poly(oxypropylene) block copolymers, also known commercially as poloxamers. Poloxamer surfactants in the present invention should have a Hydrophilic-Lipophilic

Balance (HLB) of between about 10 and about 30, and preferably between about 10 and about 25.

Amphoteric surfactants have the capacity to behave as either an acid or a base and include quaternized imidazole derivatives useful in the present invention. Preferred amphoteric agents include long chain (alkyl) amino-alkylene aklylated amine derivatives, also known as "Miranol", i.e., Miranol C_2M, or N-alkyl betaine surfactants.

Cationic surfactants are surfactants which carry a positive charge. Cationic surfactants especially useful in the present invention, include antimicrobial quaternary ammonium salts.

Fluorine-releasing compounds may be used in the dentifrice composition of this invention and may be fully or slightly water soluble.. These are chacterized by their ability to release fluoride ions or fluoride-containing ions in water and by their lack of reaction with other components in the composition.. Typical fluorine-releasing compounds are inorganic fluoride salts such as water-soluble alkali metal, alkaline earth metal, and heavy metal salts. Sodium monofluorophosphate, sodium fluoride, stannous fluoride and mixtures thereof are preferred. The most preferred fluorine-releasing compound is sodium monofluorophosphate. The amount of fluorine-releasing compound should be sufficient to provide 800-1500 ppm fluoride ion.

Sweeteners well known in the art, including natural and artificial sweeteners, may be used in the dentifrice compositions of this invention. The sweetener may be selected from a wide range of materials including naturally occurring water-soluble sweeteners, artificial water-soluble sweeteners and modified water-soluble sweeteners derived from naturally occurring water-soluble sweeteners.

In general, an effective amount of sweetener is utilized to provide the level of sweetness desired in any particular embodiment of the dentifrice compositions according to the present invention. This amount will vary with the sweetener selected and the final oral hygiene product.

The flavoring agents (flavors, flavorants) which may be used include those flavors known to the skilled artisan, such as natural and artificial flavors. Suitable flavoring agents include mints, such as peppermint, citrus flavors such as orange and lemon,

artificial vanilla, cinnamon, various fruit flavors, both singly or mixed, and the like. The amount of flavoring agent employed herein is normally a matter of preference subject to such factors as the type of final dentifrice composition, the individual flavor employed, and the strength of flavor desired.

The coloring agents (colors, colorants) useful in the present invention are used in amounts effective to produce the desired color. These coloring agents include pigments which may be incorporated in amounts up to about 6%, by weight of the composition. A preferred pigment, titanium dioxide, may be incorporated in amounts up to about 2%, and preferably less than about 1%, by weight of the composition. A full recitation of all F.D. & C colorants and their corresponding chemical structures may be found in the Kirk-Othmer Encyclopedia of Chemical Technology, 3rd Edition, in volume 5 at pages 857-884, which text is incorporated herein by reference.

Suitable abrasives in the dentifrice include hydrated silica, calcium carbonate, calcium pyrophosphate, dicalcium phosphate dihydrate, or alkali metal meta-phosphates. Silica abrasives in the dentifrice composition according to this invention may include among others, ZEODENT® (113), manufactured by J. M. Huber Corp. and SYLOID® or SYLODENT®, manufactured by W.R. Grace Co. These abrasives may be used in amounts up to about 75.0% w/w of the composition, preferably in amounts from about 5.0% w/w to about 40% w/w of the composition, and most preferably from about 7.0% w/w to about 30.0% w/w of the composition.

Suitable buffers include citric acid-sodium citrate, acetic acid-sodium acetate, sodium saccharine-acid saccharine, and benzoic acid and benzoate in amounts up to about 1%, and preferably from about 0.05% to about 0.5%, by weight of the composition. The pH for the preferred embodiment of the dentifrice of the present invention is from 3.0 to about 5.5.

Suitable preservatives in this invention include benzoic acid, sodium benzoate, butylated hydroxyanisole (BHA), butylated hydroxytoluene (BHT), ascorbic acid, methyl paraben, propyl paraben, tocopherols and mixtures thereof. Preservatives when used are generally present in amounts up to about 1.0% w/w, and preferably from about 0.1% w/w to about 1.0% w/w of the dental gel composition.

Various other materials may be incorporated in the dentifrice of this invention. Examples thereof are opacifiers, desensitizing agents, silicones and ammoniated materials such as urea, diammonium phosphate and mixtures thereof. These adjuvants are incorporated in the dentifrice in amounts which do not substantially adversely affect the desired properties and characteristics and are suitably selected and used in conventional amounts.

In the preferred embodiment form of the invention, the dentifrice composition is in the form of toothpaste or a dental gel. As used herein, the term "gel" means a solid or semisolid colloid which contains considerable quantities of water. The colloid particles in a gel are linked together in a coherent meshwork which immobilizes the water contained inside the meshwork. One skilled in the art would know how to formulate the dentifrice compositions of this invention as a past and a gel.

The present invention extends to methods of making the improved oral antiseptic compositions. The final compositions are readily prepared using methods generally known by those skilled in the art, as described in greater detail below. In such a method, an oral antiseptic dentifrice composition according to the present invention is made by first combining water, a source of zinc ion, part of the humectant, one or more sweeteners, buffers and preservatives. If fluorine-releasing compounds are used, they are added in this step. The remainder of the humectant is separately combined with one or more gums, and then combined with the first mixture. Titanium dioxide and silicas are separately mixed, and combined with the other mixture previously prepared. Finally, colors, essential oils, flavors, and surfactants are added and mixed. The pH is adjusted to a pH of about 3.0 to about 5.5 with acidifiers. A vacuum is pulled if necessary for deaeration. The pH of a 25.0% w/w aqueous solution of the composition is measured using a suitable pH meter (e.g. Orion Research Microprocessor pH/millivolt Meter, Model 811).

The apparatus useful in accordance with the present invention comprises mixing apparatus well known in the dental art, and therefore the selection of the specific apparatus will be apparent to the artisan.

The present invention is further illustrated by the following examples which are, however, not included to limit the effective scope of the claims. All parts and percentages in the examples and throughout the specification and claims are by weight of the final composition unless otherwise specified. Although the invention is described with regard to the following illustrative examples, it will be apparent to one skilled in the art that various modifications may be made thereto which fall within its scope.

EXAMPLE 1

A mouthwash with the ability to prevent the creation of malodors for several hours was prepared by combining the following components in potable water.

COMPONENTS	AMOUNT
Ethanol	216 mls.
Thymol	0.639 grams
Menthol	0.425 grams
Methyl Salicylate	0.66 grams
Eucalyptol	0.922 grams
$ZnCl_2$	1.0 grams
Benzoic Acid	1.2 grams
Sodium Benzoate	0.35 grams
Caramel	0.24 grams
Poloxamer 407	2.0 grams
Sorbitol	200 grams
1-Propanol (Flavorant)	5.0 grams
Water	Q.S. to 1 Liter

The above composition was prepared by adding the essential oils (thymol, menthol, methyl salicylate and eucalptol), Poloxamer 407, benzoic acid, and 1-propanol to ethanol, followed by the addition of 250 ml of potable water.

To that mixture was added $ZnCl_2$. Sodium benzoate, zinc chloride, sorbitol, and caramel were then added; q.s. to 1000 ml with potable water.

EXAMPLE 2

A dentifrice with zinc chloride and essential oils was formulated. Another component in the dentrifrice was hydroxyethylcellulose (Natrosol 250H.) Natrasol 250H in the dentifrice helps reduce the astringency caused by zinc ions.

Ingredient	Percent	For 1kg
Water, purified	32.52930	325.2930
Sorbitol 70%	35.000	350.0000
Polyethylene glycol	2.000	20.0000
Sodium fluoride 1100ppm	0.2430	2.430
Na Saccharin NF 000A176	0.700	7.0000
Saccharin NF 000A176	0.500	5.0000
Zinc chloride	0.500	5.0000
Sodium benzoate	0.200	2.0000
FD+C Blue #1 .2%	1.000	10.0000
D&C Yellow #10 .1%	0.250	2.5000
Sylodent 753	25.0	250.0000
Titanium dioxide	0.350	3.5000
Natrosol 250H	0.75	7.5
CMC 7mf	1.1	11.0
Glycerin 95%	6.000	60.0000
* 17814-057-1 flavor (-17)	2.36670	23.6670
SLS	1.500	15.0000
SUB TOTAL	67.4707	674.7070
TOTAL	100.0000	1000.0000
TOTAL FLAVOR	2.3667	23.6670
	pH	Viscosity
* Includes: Thymol 0.5112%, Methyl salicylate 0.4800%, Menthol 0.3400%, and Eucolyptol 0.7376%		

EXAMPLE 3

Assay for Dentifrice Antimicrobial Activities

The relative antimicrobial activity of several oral compositions of this invention was tested using an *ex vivo* kinetic kill time assay and a Plaque Kill & Growth kinetics (PKGK) assay. In the kinetic kill time assay, saliva suspensions containing natural oral organisms were vigorously mixed with a dentifrice sample in a 3.5:1 ratio. One ml aliquots of the assay mixture were removed and diluted into a neutralizing broth in a 1:9 ratio at 30 and 60 seconds, which periods represent the length of time typically spent in toothbrushing. 50 µl aliquots of diluted suspension were plated on nutrient agar plates, which were incubated anaerobically at 37° C and counted to enumerate surviving microbial colonies. Relative antiseptic activity of each dentifrice was determined as a function of the number of surviving organisms recovered on agar plates.

The PKGK assay determined the *in vivo* antibacterial efficacy of a single dentifrice use in which product was brushed onto one dental quadrant for 15 seconds, then dispersed throughout the mouth for 45 seconds using the tongue and "swishing" in a manner previously described by White *et al* (*J. Dent. Res.* **72A**, #1346, 1993; *J. Clin. Dent.* special issue **IV**: 59-71, 1995). Bactericidal and bacteriostatic effects against plaque microorganisms were assessed by 1) quantitative culturing of turbidity-adjusted plaque samples, and 2) growth kinetics analysis (lag time) of the plaque bacterial suspensions incubated in nutrient broth. Reductions in viable bacterial counts reflect bactericidal efficacy, and increases in lag time represent both nonlethal inhibitory activity as well as reductions in the viable/nonviable cell ratio. The study population consisted of 12 subjects who used all six treatments in a crossover design. Post-treatment plaque samples were collected at 1-, 2- and 3-hours after product use and analyzed for growth lag time as the primary efficacy variable and recovered bacterial number as the secondary efficacy variable.

Experimental Example 1

Dentifrice formulations of this invention were prepared using ingredients and quantities as shown in Table 1. Formulas 1.1-1.3 of this invention were formulated with increasing levels of zinc chloride plus a constant level of the essential oils thymol, menthol, methyl salicylate and eucalyptol. Formulas 2.1-2.3 of this invention were formulated with increasing levels of zinc chloride without essential oils.

Table 1. Formulations used in Experimental Example 1.

Formula Number	1.1	1.2	1.3	2.1	2.2	2.3
Zinc chloride	0	0.5	1.0	0	0.5	1.0
Thymol	0.5	0.5	0.5	0	0	0
Menthol	0.195	0.195	0.195	0	0	0
Methyl Salicylate	0.395	0.395	0.395	0	0	0
Eucalyptol	0.01	0.01	0.01	0	0	0
Sorbitol, 70%	40	40	40	40	40	40
Sodium fluoride	0.243	0.243	0.243	0.243	0.243	0.243
Sodium saccharin	0.8	0.8	0.8	0.8	0.8	0.8
Saccharin	0.4	0.4	0.4	0.4	0.4	0.4
Sodium Benzoate	0.2	0.2	0.2	0.2	0.2	0.2
FD+C Blue #1	0.002	0.002	0.002	0.002	0.002	0.002
D+C Yellow #10	0.00025	0.00025	0.00025	0.00025	0.00025	0.00025
Sylodent 753	30	30	30	30	30	30
TiO_2	0.35	0.35	0.35	0.35	0.35	0.35
Xanthan	0.5	0.5	0.5	0.5	0.5	0.5
Hydroxyethylcellulose	1.0	1.0	1.0	1.0	1.0	1.0
Glycerin	5.0	5.0	5.0	5.0	5.0	5.0
Flavors	Q.S.	Q.S.	Q.S.	Q.S.	Q.S.	Q.S.
Sodium lauryl sulfate	1.5	1.5	1.5	1.5	1.5	1.5
Water	To 100%	To 100%	To 100%	To 100%	To 100%	To 100%

Experimental Example 2

A factorial designed study was performed to determine the relationship between zinc chloride, essential oils (thymol, menthol, methyl salysalate, eucalyptol) and both antiseptic activity and bacteriostatic of dentifrice compositions using the formulations in Table 1. In this study, zinc chloride levels ranged 0%, 0.5% and 1.0% in the presence of essential oils (formulas 1.1, 1.2, 1.3) or in the absence of essential oils (formulas 2.1, 2.2, 2.3). *Ex vivo* kinetic kill time assay and *in vivo* PKGK assay were employed to evaluate all six formulations as listed in Table 1.

The results of this study clearly showed that essential oils impart high levels of antiseptic activity to the dentifrice formulas, whereas zinc chloride-containing dentifrice formulas showed only a modest antiseptic activity as demonstrated in both *ex vivo* kinetic kill time assay and *in vivo* PKGK assay.

In the *in vivo* PKGK assay, bacteriostatic efficacy of the dentifrice compositions was determined by measuring growth kinetic lag time of the plaque bacterial suspensions incubated in nutrient broth. Unexpectedly, the combination of zinc chloride and essential oils resulted in a significant increase in bacteriostasis compared to the formulas without essential oils. Furthermore, the enhancement in bacteriostasis by the addition of essential oils became more pronounced in later post-treatment phases, indicating long lasting efficacy. As shown in Figure 1, a clear dose response was observed between zinc chloride levels and bacteriostatic efficacy in the presence of essential oils (formulas 1.1, 1.2 and 1.3). In the absence of essential oils, bacteriostatic effects of zinc chloride were only observed at 1-hour post-treatment time point. At 2- and 3-hour post-treatment time points, no significant bacteriostatic efficacy was observed (formulas 2.1, 2.2 and 2.3).

What is claimed is:

1. An improved oral composition comprising a bacteriostatic and anticalculus effective amount of zinc ion provided by one or more zinc-containing compounds and an antimicrobial effective amount of one or more essential oils.

2. The improved oral composition of claim 1 wherein said oral composition is a dentifrice.

3. The improved oral composition of claim 2 wherein the amount of zinc ion in said composition is at least about 50 ppm.

4. The improved oral composition of claim 3 wherein the amount of zinc ion in said composition is at least about 1500 ppm.

5. The improved oral composition of claim 2 wherein said one or more zinc-containing compounds are selected from the group comprising zinc salts and zinc oxide.

6. The improved oral composition of claim 5 wherein said one or more zinc salts are selected from the group comprising zinc chloride, zinc sulphate, zinc bromide, zinc iodide, zinc nitrate, and carboxylic acid salts of zinc.

7. The improved oral composition of claim 6 wherein said zinc salt is zinc chloride.

8. The improved oral composition of claim 6 wherein said one or more zinc salts are present in an amount of at least about 0.01% by weight of said composition.

9. The improved oral composition of claim 8 wherein said one or more zinc salts are present in an amount of at least about 0.2% by weight of said composition.

10. The improved oral composition of claim 9 wherein said one or more zinc salts are present in an amount of at least about 0.5% by weight of said composition.

11. The improved oral compositon of claim 1 wherein said one or more essential oils are selected from the group comprising thymol, menthol, methyl salicylate, and eucalyptol.

12. The improved oral composition of claim 11 wherein said one or more essential oil is thymol.

13. The improved oral composition of claim 12 wherein the concentration by weight of thymol is in an amount of from about 0.01% w/w to about 1.0% w/w; the concentration by weight of menthol is from about 0.01% w/w to about 1.0% w/w; the

concentration by weight of eucalyptol is from about 0.01% w/w to about 1.0% w/w; and the concentration of methyl salicylate is from about 0.01% w/w to about 1.0% w/w.

14. The improved oral composition of claim 1 wherein said oral composition is a mouthwash.

15. A method for preparing an improved dentifrice comprising a bacteriostatic and anticalculus effective amount of zinc ion provided by one or more zinc-containing compounds and an antimicrobial effective amount of one or more essential oils comprising:

a) combining water, a source of zinc ion, one or more humectant, sweeteners, buffers and preservatives;

b) admixing one or more humectant with gum to form a second mixture;

c) admixing said first and second mixture to form a third mixture;

d) admixing titanium dioxide and silicas to form a fourth mixture;

e) admixing said third and fourth mixtures to form a fifth mixture

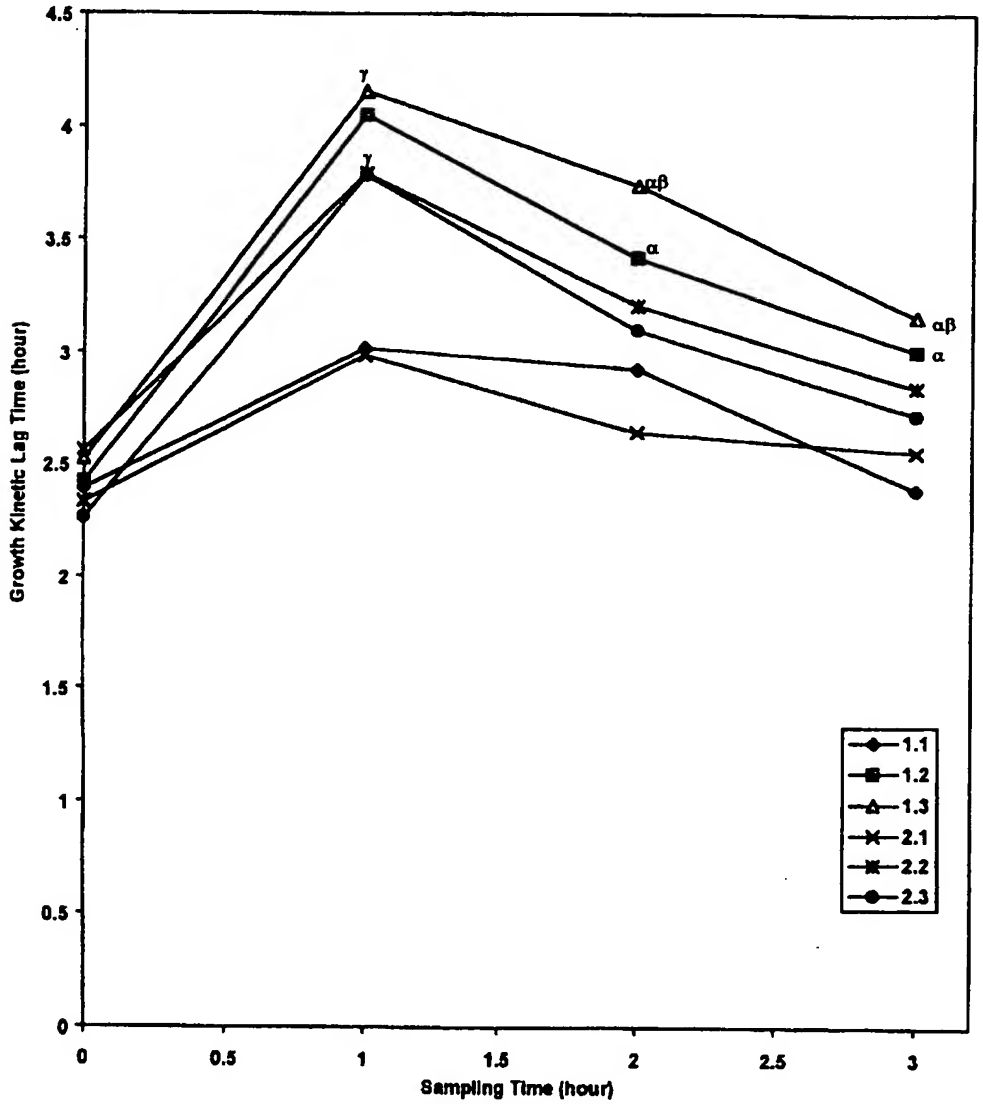
f) admixing said fifth mixture with colors, essential oils, flavors, and surfactants to form a sixth mixture;

g) adjusting the pH of said sixth mixture to a pH of about 3.0 to about 5.5 with acidifiers;

h) dearating siad sixth mixture under a vacuum.

16. A method or reducing oral malodor by using an improved oral composition comprising a bacteriostatic and anticalculus effective amount of zinc ion provided by one or more zinc-containing compounds and an antimicrobial effective amount of one or more essential oils.

Figure 1. Bacteriostatic Efficacy Determined by *In Vivo* PKGK Assay



α: P<0.05 compared with 1.1 at the same post-treatment time point.
β: P<0.05 compared with 2.3 at the same post-treatment time point.
γ: P<0.05 compared with formulas 1.1 or 2.1 at 1-hour time point.

INTERNATIONAL SEARCH REPORT

Interr ial Application No
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A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 A61K7/16 A61K7/26

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 A61K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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X	US 1 488 097 A (CREGER) 25 March 1924 see claims 2-5; example 1	1-14,16
X	US 5 405 603 A (MACKLES ET AL.) 11 April 1995 see claims 1,5,7,12; example 14	1-14,16
X	US 5 407 662 A (MACKLES ET AL.) 18 April 1995 see example 6	1-14,16

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[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

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Date of the actual completion of the international search	Date of mailing of the international search report
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Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+ 31-70) 340-2040, Tx. 31 651 epo nl, Fax (+ 31-70) 340-3016	Fischer, J.P.

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Inter nal Application No
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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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